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SPECIFICATION FOR ALUMINA, CHROMATOGRAPHIC GRADE

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INDIAN STANDARDS INSTITUTION
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

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SPECIFICATION FOR ALUMINA, CHROMATOGRAPHIC GRADE

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Indian Standard

SPECIFICATION FOR ALUMINA, CHROMATOGRAPHIC GRADE

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 28 January 1985, after the draft finalized by the Inorganic Chemicals (Miscellaneous) Sectional Committee had been approved by the Chemical Division Council.

0.2 Alumina is manufactured by precipitation technique. Precipitation is achieved by close control of operating parameters, such as *p*H, concentration and temperature. For preparation of chromatographic grade alumina high purity raw materials are used and care is taken during processing to achieve product purity. The material is represented by an empirical formula $Al_2O_3.nH_2O$.

0.3 Chromatographic grade alumina is used for preparative column chromatography and analytical thin layer chromatography.

0.4 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for chromatographic grade alumina.

2. TYPES

2.1 The material shall be of the following three types:

a) Acidic type,

b) Neutral type, and

c) Basic type.

---

*Rules for rounding off numerical values (*revised*).

3. REQUIREMENTS

3.1 Description — The material shall consist of aluminium oxide in granular form and may be supplied in the following size ranges:

a) 250-180 micron IS Sieve,

b) 180-150 micron IS Sieve,

c) 150-125 micron IS Sieve, and

d) 125-75 micron IS Sieve.

3.2 The material shall comply with the requirements given in Table 1 when tested according to the methods prescribed in Appendix A. Reference to the relevant clauses of Appendix A is given in col 6 of the table.

TABLE 1 REQUIREMENTS FOR ALUMINA, CHROMATOGRAPHIC GRADE

Sl No.	Characteristic	Requirement for Type			Method of Test (Ref to Cl No. in Appendix A)
		Acidic	Neutral	Basic	
(1)	(2)	(3)	(4)	(5)	(6)
i)	Bulk density g/ml, *Min*	0·9	0·9	0·9	A-2
ii)	*p*H value	3·5-4·5	6·5-7·5	9·0-10·0	A-3
iii)	Sodium (as Na_2O), percent by mass, *Max*	0·5	0·5	0·5	A-4
iv)	Chlorides (as Cl), percent by mass, *Max*	0·08	0·03	0·03	A-5
v)	Sulphates (as SO_4), percent by mass, *Max*	0·1	0·1	0·1	A-6
vi)	Loss on ignition, *Max*	3·0	4·0	2·0	A-7
vii)	Brockman activity	Between II and III at 40-65 percent relative humidity			A-8

3.2.1 *Additional Requirements* — If required by the purchaser, the material shall contain not more than 100 mg/l of ammonia and/or ammonium compounds, as tested by the method given in **A-9**.

4. PACKING AND MARKING

4.1 Packing — The material shall be packed in clean, dry and air-tight bottles. The container shall not be opened until required for use.

4.2 Each container shall bear legibly and indelibly the following information:

a) Name, type and size of the material;
b) Name of the manufacturer and/or his recognized trade-mark, if any;
c) Mass of the material in the container;
d) Date of packing; and
e) Batch number.

4.2.1 The containers may also be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act, and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

5. SAMPLING

5.1 The method of drawing representative samples of the material, number of tests to be performed and the criteria for conformity of the material to the requirements of this specification shall be as prescribed in Appendix B.

APPENDIX A

(*Clause* 3.2)

METHODS OF TEST FOR ALUMINA, CHROMATOGRAPHIC GRADE

A-1. QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1977*) shall be used in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

---

*Specification for water for general laboratory use (*second revision*).

A-2. BULK DENSITY

A-2.1 Procedure — Take the material exposed to an atmosphere of 95 percent relative humidity over night and fill it in a 250-ml measuring cylinder up to 50 ml without knocking. Shake the sample down by gently lifting the cylinder as far as 5 cm from the base pad and releasing it on the table. Repeat this knocking for 4 more times. Add further 50 ml of the sample and knock five times, as above, after every 50 ml addition up to 250 ml. Level the cylinder with the material without any further knocking. Empty out the material from the cylinder and weigh to the nearest 0·5 g.

A-2.2 Calculation

$$\text{Bulk density, g/l} = 4\,M$$

$$\text{g/ml} = \frac{4\,M}{1\,000}$$

where

M = mass in g of the material in the cylinder.

A-3. *p*H VALUE

A-3.1 Apparatus

A-3.1.1 *pH Meter*

A-3.2 Procedure — Prepare an aqueous extract of the material by shaking for 1 hour, 5 g of the material with 50 ml of water (free from ammonia and carbon dioxide), in a clean polyethylene flask. Decant the clear solution and measure the *p*H by using any standard *p*H meter.

A-4. SODIUM AND ITS COMPOUNDS

A-4.1 Apparatus

A-4.1.1 *Flame Photometer*

A-4.2 Reagent

A-4.2.1 *Concentrated Nitric Acid — See* IS : 264-1976*.

A-4.3 Procedure — Weigh 0·5 g of the sample into a 250-ml glass dish previously washed with dilute nitric acid. Add 10 ml of water and 10 ml of concentrated nitric acid and evaporate the contents to dryness

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*Specification for nitric acid (*second revision*).

on a hot plate. Add another 10 ml of concentrated nitric acid and evaporate to dryness again. Extract the residue with 20 ml of nitric acid (1 : 1) by gentle boiling. Filter through Whatman No. 40 or equivalent filter paper into a 250-ml volumetric flask. Wash three times with hot water.

Cool the flask and dilute to the mark with water. Take 25 ml of the solution and dilute to 100 ml in a volumetric flask. Flame the diluted solution using a flame photometer. The instrument shall be calibrated in the range of 0 to 5 mg/l of sodium.

A-4.4 Calculation

Sodium and its compounds (as Na_2O), percent by mass = $X \times 0{\cdot}2$

where

X = mg/l of Na_2O from the calibration curve.

A-5. WATER SOLUBLE CHLORIDES

A-5.1 Reagents

A-5.1.1 *Dilute Nitric Acid* — 50 percent (*m/v*).

A-5.1.2 *Standard Silver Nitrate Solution* — 0·05 N.

A-5.1.3 *Standard Ammonium Thiocyanate Solution* — 0·05 N.

A-5.1.4 *Ferric Alum Indicator Solution* — 10 percent (*m/v*).

A-5.2 Procedure

A-5.2.1 *Preparation of Solution* — Weigh accurately 2 g of the material in a platinum crucible of 40 ml capacity previously washed with nitric acid. Heat the crucible and its contents in a furnace at 350°C for 45 minutes. Transfer the contents to a 250-ml beaker and add about 100 ml of water. Boil for 2 to 3 minutes and then digest for half-an-hour. Allow to settle for 10 minutes and filter through Whatman No. 40 filter paper or equivalent into a 200-ml standard flask; wash 3 times with hot water. Cool the solution and make up to the mark.

A-5.2.2 Take 100-ml aliquot of the prepared solution (*see* **A-5.2.1**) in a 400-ml conical beaker and acidify with 1 ml of dilute nitric acid. Add enough water to make up the volume to about 100 ml. Cool the beaker and contents. Add 10 ml of standard silver nitrate solution and back titrate against standard ammonium thiocyanate solution to reddish brown end-point using 5 to 10 drops of ferric alum indicator. Take a blank reading by adding 10 ml of standard silver nitrate to about 100 ml of water and back titrating against standard ammonium thiocyanate solution using ferric alum as indicator.

A-5.3 Calculation

$$\text{Water soluble chlorides (as Cl), percent by mass} = \frac{(V_1 - V_2) \times 10 \times 0{\cdot}001\,775 \times 100}{V_1}$$

where

V_1 = volume in ml of standard ammonium thiocyanate solution required for the blank, and

V_2 = volume in ml of standard ammonium thiocyanate solution required for titration of the aliquot taken for the test.

A-6. WATER SOLUBLE SULPHATES

A-6.1 Reagents

A-6.1.1 *Dilute Hydrochloric Acid* — 50 percent (*m*/*v*).

A-6.1.2 *Barium Chloride Solution* — 10 percent (*m*/*v*).

A-6.2 Procedure — Take 100 ml of the prepared solution (*see* **A-5.2.1**) in a 400-ml beaker. Acidify with 1 to 2 ml of dilute hydrochloric acid and heat to boiling. Add 10 ml of hot barium chloride solution drop by drop until all the sulphate is precipitated. Digest on a water bath until the precipitate has settled or preferably keep overnight, filter through a filter paper (Whatman No. 44 or its equivalent) and wash with hot water until washings are free from chlorides. Ignite the precipitate in a tared platinum crucible at 900°C for 30 minutes, cool in a desiccator and weigh.

A-6.3 Calculation

$$\text{Water soluble sulphates, percent by mass} = M \times 41{\cdot}16$$

where

M = mass in g of ignited residue.

A-7. LOSS ON IGNITION

A-7.1 Apparatus

A-7.1.1 *Platinum Crucible*

A-7.2 Procedure — Take 1 g of the material in a clean, dry platinum crucible. Keep the crucible and its contents on a hot plate for 10 minutes. Heat further at 550°C for 5 minutes and finally at 1 100°C for 30 minutes. Cool, weigh and calculate the percent loss on ignition.

A-8. BROCKMAN ACTIVITY

A-8.1 Reagents

A-8.1.1 *Benzene*

A-8.1.2 *p-Aminoazobenzene Dye in Benzene* — 5 percent.

A-8.2 Apparatus

A-8.2.1 *Capillary Tube* — melting point type, 80 mm × 1 mm closed at one end.

A-8.3 Procedure — Grind the sample to 75-micron IS Sieve and expose it to 50 ± 5 percent relative humidity for about 1 hour. Pack the capillary tube uniformly with the sample (by tapping it from time to time during packing) so that no voids are left inside the sample. Break open the sealed end of the tube. Put, through the broken end, a drop of dye solution over the sample and invert the tube in a beaker/flask containing benzene, so that the dye-end is immersed in benzene. The dye will move upwards as benzene reaches the top of the sample forming a band. Determine the height through which the centre of the band rises in the tube, using a graduated scale.

A-8.4 Calculation — If h is the height (in mm) through which dye rises in the capillary, then resolution factor is given by $rf = h/80$. Relation between rf and Brockman Activity is as follows:

rf	0	0·25	0·30	0·43	0·55
Activity	I	II	III	IV	V

A-9. AMMONIA AND AMMONIUM COMPOUNDS

A-9.1 Reagents

A-9.1.1 *Potassium Sodium Tartrate* (*Rochelle Salt*) — 50 percent solution. Dissolve 50 g of the hydrated salt in water and dilute to 100 ml.

A-9.1.2 *Nessler Reagent* — Dissolve 3·5 g of potassium iodide and 1·25 g of mercuric chloride in 80 ml of water. Add a cold saturated aqueous solution of mercuric chloride, stirring constantly, until a slight red precipitate remains, then add 12 g of sodium hydroxide. Allow this to dissolve, add a little more of the saturated mercuric chloride solution until a slight turbidity is obtained and dilute to 100 ml with water. Allow to settle, decant and store in the dark place.

A-9.1.3 *Standard Ammonia and Ammonium Compounds Solution* — Dissolve 0·031 4 g of ammonium chloride in water and dilute to 1 000 ml. One millilitre of the resulting solution = 10 µg of NH_3. The solution shall be freshly prepared.

A-9.2 Procedure — Dilute 25 ml of an aqueous extract prepared as in **A-3.2** to 50 ml with water, add 1 ml of the potassium sodium tartrate solution and 1 ml of Nessler reagent, mix well and allow to stand for 10 minutes. At the same time, treat similarly 2·5 ml of the standard ammonia and ammonium compounds solution (*see* **A-9.1.3**).

Reject if any colour produced in the test solution is stronger than that produced in the standard.

APPENDIX B

(*Clause* 5.1)

SAMPLING OF ALUMINA, CHROMATOGRAPHIC GRADE

B-1. GENERAL REQUIREMENTS OF SAMPLING

B-1.1 The sampled material shall be kept in a protected place and shall not be exposed to damp air.

B-1.2 The samples shall be placed in suitable containers and each container shall be marked with full details of sampling giving the date of sampling; type of material, batch number, if any; and the name of the manufacturer.

B-2. SCALE OF SAMPLING

B-2.1 All the containers in a single consignment of the material of same grade drawn from a single batch of manufacture shall constitute a lot.

B-2.2 Samples shall be tested from each lot for ascertaining conformity of the material to the requirements of the specification.

B-2.3 The number of containers *n* to be chosen from the lot shall depend on the size of the lot *N* and shall be as given in Table 2.

B-2.4 The containers to be selected for sampling shall be chosen at random from the lot. For this purpose a random number table shall be used (*see* IS : 4905-1968*). In case such a table is not available, the following procedure shall be adopted:

> 'Starting from any container, count them as 1, 2, 3, ...,*r* and so on in a systematic manner, where *r* is the integral part of *N*/*n*. Every *r*th container thus counted shall be withdrawn to constitute the required sample.'

TABLE 2 SCALE OF SAMPLING

(*Clause* B-2.3)

LOT SIZE N (1)	NO. OF CONTAINERS TO BE SELECTED n (2)
Up to 15	3
16 to 50	5
51 to 100	8
101 and above	13

B-3. PREPARATION OF SAMPLE

B-3.1 From each of the containers selected, draw approximately equal quantity material with the help of suitable sampling implement. The material drawn from different containers shall be mixed thoroughly to give a composite sample which shall be sufficient to make triplicate determination for all the characteristics given in this specification.

B-3.2 The composite sample shall then be divided into three parts, for the purchaser, another for the supplier and the third for the referee. These parts shall be transferred to separate containers which shall be suitably closed and marked with all the details of sampling.

B-3.3 The referee sample bearing the seals of both the purchaser and the supplier shall be kept at a place agreed to between the two. It shall be used in case of a dispute between the two.

B-4. NUMBER OF TESTS AND CRITERIA FOR CONFORMITY

B-4.1 Tests for all the characteristics given in **3** shall be conducted on the composite sample.

B-4.2 The lot shall be considered as conforming to the requirements of the specification if all the test results on the composite sample satisfy the corresponding requirements given in **3**. The Brockman activity test (Table 1) should be considered the most important of all the other tests.

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*Methods for random sampling.

INTERNATIONAL SYSTEM OF UNITS (SI UNITS)

Base Units

QUANTITY	UNIT	SYMBOL
Length	metre	m
Mass	kilogram	kg
Time	second	s
Electric current	ampere	A
Thermodynamic temperature	kelvin	K
Luminous intensity	candela	cd
Amount of substance	mole	mol

Supplementary Units

QUANTITY	UNIT	SYMBOL
Plane angle	radian	rad
Solid angle	steradian	sr

Derived Units

QUANTITY	UNIT	SYMBOL	DEFINITION
Force	newton	N	$1\ N = 1\ kg.m/s^2$
Energy	joule	J	$1\ J = 1\ N.m$
Power	watt	W	$1\ W = 1\ J/s$
Flux	weber	Wb	$1\ Wb = 1\ V.s$
Flux density	tesla	T	$1\ T = 1\ Wb/m^2$
Frequency	hertz	Hz	$1\ Hz = 1\ c/s\ (s^{-1})$
Electric conductance	siemens	S	$1\ S = 1\ A/V$
Electromotive force	volt	V	$1\ V = 1\ W/A$
Pressure, stress	pascal	Pa	$1\ Pa = 1\ N/m^2$